

CHARACTERIZATION OF A SHALE OIL PRODUCED BY IN SITU RETORTING

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INTRODUCTION

In situ or in place retorting has many potential advantages over the more conventional mining/retorting approach. One of the major advantages is that in situ retorting avoids the costly and disfiguring mining and spent-shale disposal steps associated with the mining/retorting approach. Another potential advantage is that it may make it possible to recover oil from formations that are not amenable to the mining/retorting approach. Many investigators have recounted at length the problems of in situ retorting and Burwell, Sterner, and Carpenter¹ have adequately referenced these investigations. Whereas it is not the purpose of this paper to delve into extended discussion of in situ retorting, it may help the reader to have an idea of the conditions under which in situ oils are produced. This paper will, therefore, describe briefly an in situ experiment and a brief description will also be made of each of the retorts that produced the oils with which the in situ oils will be compared.

This paper will describe two crude shale oils that were recovered from successful in situ-combustion retorting experiments.¹ The description will be based primarily on the results of the application of the Bureau of Mines crude shale-oil analysis² to these two crudes. In addition to describing the properties of the two in situ crudes in terms of this analysis, the properties of crude shale oils from aboveground retorts will also be described. Comparison of the oils from the aboveground retorts with each other will be made in order to determine the effect of change of retorting parameters upon oil character. Comparison of the in situ oils with the aboveground-retort oils will allow explanation of the retorting conditions under which the in situ oils were produced. This indirect approach to the problem of describing the conditions under which in situ retorting is accomplished is necessary because of the inaccessibility of the retort zone to visual inspection, either during or after retorting.

DESCRIPTION OF RETORTS

Aboveground Retorts

Two types of aboveground retorts featuring internal combustion for heating purposes will be discussed. The main difference in these two types, as far as this paper is concerned, is in the size of shale that is charged. The gas combustion retort is a type that uses crushed, screened, small-size shale and that accomplishes retorting with short, rapid shale heatup. The 10-ton retort and the 150-ton retort are of the type that uses mine-run shale and that accomplishes retorting with a long, slow shale heatup. Thus gas combustion retorting is done rapidly at relatively high temperatures compared with the retorting done in the type of retorts that use mine-run shale because in gas combustion retorting the shale does not undergo a long, low-temperature period of soaking. Table 1 lists the size of shale and the combustion-zone velocity for the aboveground retorting experiments.

Gas Combustion Retort. --The gas combustion retort is characterized by the use of continuous gravity flow of crushed, screened shale; direct gas-to-solids heat exchange; and heat supply by internal combustion. The retort is a vertical, refractory-lined vessel equipped with shale- and

TABLE 1. - Raw-shale size distribution and velocity of combustion zone

Retort	Source of shale	Wt pct of raw shale Raw shale size, inches				Combustion zone velocity, ft/day
		Less than 4	4 to 10	10 to 20	Greater than 20	
Gas comb. ^{1/}	^{2/} Colo.	^{3/} 100	0	0	0	200
10-Ton	^{2/} Colo.	50	20	15	15	2
10-Ton	Wyo.	35	35	20	5	2
150-Ton	Colo.	40	20	20	20	2

^{1/} Gas combustion.

^{2/} Colo. = Anvil Points, Colorado; Wyo. = Rock Springs, Wyoming.

^{3/} All shale minus 3-inch plus 1/4-inch.

gas-distributing devices. The combustion air and retort products move countercurrently to the shale. Incoming shale serves to cool the products of retorting and is in turn rapidly heated to retorting temperature by the gases. The hot, retorted shale preheats the recycle gas before the recycle gas enters the combustion zone in the retort. In the combustion zone, the heat for retorting is produced by burning some of the organic residue on the shale plus recycle gas. A complete description of the gas combustion retort and its operation is given in a Bureau of Mines publication by Ruark, et al.³

10-Ton Retort. --The 10-ton retort was built and operated to study the retorting characteristics of mine-run shale having pieces as large as 20 inches on each side.⁴ The retort is a batch unit consisting of a cylindrical steel shell 6 feet inside diameter by 12 feet tall. This shell surrounds a tapered refractory lining 6 inches thick at the bottom and 9 inches thick at the top. A 1-inch steel plate that is perforated with 3/8-inch holes is used as a grate.

In practice, the retort is loaded with mine-run shale and the bed of shale is ignited at the top with a natural-gas burner. After ignition the burner is shut down. Subsequently air and recycle gas are fed to the top of the retort to maintain combustion and to accomplish retorting by heating the bed of shale from the top downward with the hot combustion gases. Control of the combustion-zone velocity is accomplished by adjusting air and recycle-gas feed rates.

150-Ton Retort. --In principles of construction and operation the 150-ton retort is similar to the 10-ton retort.⁵ It was built primarily to study the retorting characteristics of ungraded shale containing blocks of shale even larger than would fit into the 10-ton retort. The 150-ton retort is 11-1/2 feet inside diameter by 45 feet high. For the run which produced the 150-ton retort oil described in this report, the mine-run raw shale contained blocks of shale measuring as large as 3 feet x 4 feet x 5 feet and weighing as much as 3-3/4 tons.

In Situ Retort

The two in situ oils were produced during in situ combustion experiments conducted in the Green River oil-shale formation near Rock Springs, Wyoming.¹ One objective of these experiments was to study the engineering problems associated with the establishment, maintenance, and control of a self-sustaining combustion zone in previously fractured oil shale. A second objective was to produce shale oil using such a self-sustaining combustion zone. Both combustion experiments were successful in meeting their objectives. The following description is for the Bureau's first in situ experiment¹ near Rock Springs, Wyoming at a location designated as site 4. The

experimental conditions for the second in situ experiment at a site 7 will not be described because they were similar to those at site 4.

For the site 4 experiment, a 20-foot thick section of oil shale, located from 68 to 88 feet below the surface, was ignited by use of a propane burner in the center well of an expanded, five-spot development pattern. Four wells of the five-spot pattern were located on the corners of a square 25 feet on each side; the fifth well was in the center of the square. After combustion was established, the propane burner was removed and air was injected into the center well. The combustion zone was maintained in the formation for 6 weeks, at which time air injection was terminated and the combustion phase of the experiment was brought to a close. The outlying or observation wells of the expanded five-spot pattern were the wells from which flowed the unused air, the combustion products, and the retort products. Several thousand gallons of shale oil were recovered from the experiment. The oil described in this paper was produced during the last week of the experiment and is considered a representative, "steady-state" oil. Site geology, site preparation, instrumentation, pre-ignition fracturing, ignition, and retorting have been described in detail by Burwell, et al.¹

OIL CHARACTERIZATION PROCEDURES

Bureau of Mines Crude Shale-Oil Analysis.--A complete description of this analysis method has been reported by Stevens, et al.² Basically, the method tests a crude shale oil for the properties listed in table 2. In addition, specific gravity and total nitrogen content are determined on the following distillation fractions: Naphtha (IBP to 400° F), light distillate (400 to 600° F), light gas oil (600 to 800° F), and a distillation residuum (above 800° F). The naphtha and light distillate fractions are further tested for tar acid, tar base, saturate, olefin, and aromatic contents.

TABLE 2. - Properties of crude shale oils

Retort	Source of shale	Oil properties				
		Specific gravity, 60°/60° F	Pour point, °F	Viscosity, 100° F SUS	Nitrogen, wt pct	Sulfur, wt pct
Gas comb.	Colo.	0.942	80	370	1.46	0.77
10-Ton	Colo.	.923	60	112	1.57	.79
10-Ton	Wyo.	.920	50	112	1.27	.59
150-Ton	Colo.	.909	60	98	1.59	.94
In situ-4 ^{1/2}	Wyo.	.885	40	78	1.36	.72
In situ-7 ^{2/3}	Wyo.	.899	30	52	1.84	.78

^{1/} Site 4, Rock Springs, Wyoming, in situ experiment.

^{2/} Site 7, Rock Springs, Wyoming, in situ experiment.

Simulated Distillation.--This analysis method employs a nonpolar, temperature-programmed gas liquid chromatographic system to obtain an estimation of the distillation range of crude oils. The resulting simulated-distillation (sd) data can be used to predict the amount of each distillate in the crude as weight percent of the crude. The sd parameters used to obtain the crude oil compositions reported in this paper are as follows: The column was 0.180 inch inside diameter x 18 inches long; the packing was 5 percent w/w UCW-98 on 70/80 mesh Chromosorb W, acid and base washed; the temperature was programmed to give a normal triacontane emergence time of 30 minutes; and flame-ionization detection was used.

The sd weight percent data were converted to the volume percent data shown in table 3 by using the observed specific gravities of the three, lowest boiling fractions and by using estimated gravities for the highest boiling fraction and for the residuum. These estimations took into account: (1) The relative amounts of heavy gas oil and residuum from sd and (2) the observed specific gravity of the residuum from the above crude shale-oil analysis.

TABLE 3. - Crude oil composition

Retort	Source of shale	Fraction, vol pct of crude ^{1/}				
		Naphtha, IBP to 400° F	Light distillate, 400° to 600° F	Light gas oil, 600° to 800° F	Heavy gas oil, 800° to 1,000° F	Residuum, over 1,000° F
Gas comb.	Colo.	5.6	18.9	25.4	27.5	22.7
10-Ton	Colo.	5.7	26.2	33.3	26.2	8.6
10-Ton	Wyo.	5.0	29.7	36.0	24.4	4.9
150-Ton	Colo.	6.5	30.9	35.6	20.4	6.6
In situ-4	Wyo.	14.9	48.3	24.0	6.2	6.6
In situ-7	Wyo.	11.2	41.5	29.6	12.3	5.4

^{1/} Determined by adjusting simulated-distillation data for specific gravity of the fractions.

RESULTS AND DISCUSSION

Table 1 lists the size of shale and the combustion-zone velocity for the aboveground retorting experiments. The four experiments listed in the table were chosen to demonstrate effect of the following parameters on shale-oil character: (1) Rapid retorting of small-size shale, (2) slow retorting of mine-run shale, (3) slow retorting of mine-run shale with added large blocks, and (4) shale source. Gas-combustion retorting of Colorado shale demonstrates the effect of rapidly retorting crushed and screened small shale. The experiment in which Colorado shale was charged to the 10-ton retort demonstrates the effect of slowly retorting mine-run shale. Wyoming shale charged to the 10-ton retort demonstrates the effect of slowly retorting shale from a different source. The 150-ton retort experiment demonstrates the effect of slowly retorting larger mine-run shale than was retorted in the 10-ton experiments. Table 2 lists some properties of these four oils and also the properties of the two oils produced by the two in situ combustion experiments, and table 3 lists the distillation yields from each of the six crude oils.

The effects of shale size, combustion-zone velocity, and effective retorting temperature are shown by comparing the properties of the oil from the rapid retorting of small shale (gas-combustion retort) with those of oils from the slow retorting of large shale (10-ton and 150-ton retorts). In general the naphtha, light distillate, and light gas oil contents increase and the heavy gas oil and residuum contents decrease as the retorting rate and temperature become lower and the shale larger. If the foregoing parameters are the ones that have a marked influence on the properties of the oils from the aboveground retorts, then the properties of the two in situ oils can be used to demonstrate the relative value of these parameters for in situ retorting. The in situ crude oils have even higher contents of light components (naphtha and light distillate) and lower contents of heavy components (light gas oil, heavy gas oil, and residuum) than do the oils from the slow, low-temperature, aboveground retorting of mine-run shale. Thus the in situ retorting was likely accomplished at lower temperatures, at slower combustion-zone velocities, and on larger or at least a greater proportion of large shale blocks than were used in the aboveground retorting experiments.

The crude-oil composition (table 3) and the crude oil yields for the aboveground oils (column 3, table 4) can be used to calculate the amount of each distillate that is produced from each ton of 30-gallon-per-ton (gpt) oil shale. The calculated data in table 4 (columns 4 through 8) show that for each of the four aboveground experiments the combined quantity of naphtha and light distillate is about the same and amounts to about 6.9 gpt of 30-gpt shale. The quantity of light gas oil also remains constant at about 7 gpt of 30-gpt shale. However, the quantities of heavy gas oil and of residuum are shown to be markedly dependent upon the retorting parameters of shale size, combustion-zone velocity, and retorting temperature. The quantity of heavy gas oil from the gas-combustion experiment is 7.4 gpt, from the two 10-ton experiments it is 5.5 gpt, and from the 150-ton it is 3.7 gpt. The quantity of residuum shows an even greater decrease with change in the retorting parameters. Residue production from the gas combustion retort amounts to 6.1 gpt, whereas from the 10-ton and 150-ton retorts it is only 1.3 gpt. The diminution of heavy oils from those retorts which utilize large blocks of shale without an accompanying increase in light oils indicates that little secondary cracking takes place in any of the large-shale, slow-retorting systems. Indeed it appears that there is some sort of distillation being carried out in which the heavy components are deposited on cold raw shale to be consumed later by the combustion zone when it passes through the area of deposition. Any secondary cracking of this heavy material should yield an increased quantity of the light oils, unless the cracking is such that gas and coke are the only products. If this is so, the net result is the same as burning the heavy components without cracking because the gases would be burned with the recycle gas and the coke would be burned when the combustion zone traverses the area of deposition.

TABLE 4. - Distillate yields for aboveground retorts

Retort	Source of shale	Yield, gpt of 30-gpt raw shale					
		Crude oil ^{1/}	Naphtha	Light distillate	Light gas oil	Heavy gas oil	Residuum
Gas comb.	Colo.	27.0	1.5	5.1	6.9	7.4	6.1
10-Ton	Colo.	20.5	1.2	5.4	6.8	5.4	1.7
10-Ton	Wyo.	22.9	1.1	6.8	8.2	5.6	1.1
150-Ton	Colo.	18.0	1.2	5.5	6.4	3.7	1.2

^{1/} Obtained from the oil recoveries for each experiment.

If it is postulated that in situ retorting produces the same quantity of naphtha plus light distillate from each ton of shale averaging 30 gpt as did the aboveground retorts, namely 6.9, then the compositional data listed in table 3 for the two in situ oils can be used to calculate approximate quantities of each of the fractions. These quantities will add together to give total oil recovery in terms of gpt of 30-gpt shale and thus give an estimation of oil yields for each of the two experiments. Table 5 gives the results of these calculations and indicates that the experiment on site 4 had a yield of 10.9 gpt of 30-gpt shale and the experiment on site 7 had a yield of 13.1 gpt of 30-gpt shale. These calculate to Fischer assay recoveries of 36 percent and 44 percent respectively from the shale in the retorted zone.

The objective of the two experiments in the 10-ton retort was to demonstrate the effect of shale source on oil properties. The crude oil properties (table 3), the crude oil composition (table 4), and the distillate yields (table 5) show very little difference in properties of the oils from the two 10-ton experiments each using shale from a different source. Hence the differences in properties among oils from the three aboveground retorts and from the two in situ experiments can logically be said to be due to retorting parameters rather than shale source.

TABLE 5. - Calculated distillate yields for in situ experiments

Experimental site	Yield, gpt of 30-gpt raw shale				Crude oil	Yield, vol pct of Fischer assay
	Naphtha plus light distillate	Light gas oil	Heavy gas oil	Residuum		
In situ-4	6.9	2.6	0.7	0.7	10.9	36
In situ-7	6.9	3.9	1.6	.7	13.1	44

The Bureau of Mines crude shale-oil analysis tests the naphtha and light distillate fractions for saturate, olefin, and aromatic contents. Table 6 and table 7 list this analysis for these fractions from all of the six crude oils. The concentration of aromatics remains constant for each fraction. It was previously shown that for aboveground retorting the quantity of naphtha and light distillate in gpt of 30-gpt shale also was fairly constant. Therefore the quantity of aromatics is independent of the retorting parameters, and the combined quantity of saturates and olefins is also independent of the retorting parameters. However, for aboveground retorting the ratio of saturate quantity to olefin quantity is shown to be dependent upon the parameters of shale size, combustion-zone velocity, and retorting temperature. As the shale size becomes larger and combustion-zone velocity and retorting temperature become smaller, the ratio of saturates to olefins becomes larger. If this increase in saturates in the aboveground experiments is due to the slow retorting of large blocks of shale, then the in situ oils demonstrate that the in situ retorting was accomplished on larger shale and at slower rates than was the aboveground retorting.

TABLE 6. - Hydrocarbon-type analysis of neutral naphtha

Retort	Source of shale	Hydrocarbon type, vol pct		
		Saturates	Olefins	Aromatics
Gas comb.	Colo.	30	50	20
10-Ton	Colo.	34	44	22
10-Ton	Wyo.	37	42	21
150-Ton	Colo.	41	37	22
In situ-4	Wyo.	61	16	23
In situ-7	Wyo.	57	16	27

TABLE 7. - Hydrocarbon-type analysis of neutral light distillate

Retort	Source of shale	Hydrocarbon type, vol pct		
		Saturates	Olefins	Aromatics
Gas comb.	Colo.	31	41	28
10-Ton	Colo.	38	37	25
10-Ton	Wyo.	36	35	29
150-Ton	Colo.	49	28	23
In situ-4	Wyo.	55	15	30
In situ-7	Wyo.	50	22	28

Hill⁶ and Dougan⁷ have reported the characteristics of a noncombustion in situ shale oil that was produced by injection of hot natural gas at controlled temperature. In most respects the character of their crude oil is similar to that of our combustion in situ oils. Table 8 lists the distillate yields from their noncombustion in situ oil, the yields from our two combustion in situ oils, and the yields from gas-combustion oil. Their combined naphtha and light distillate fractions account for 80 percent of their in situ crude. Our two in situ oils have 60 percent from the site 4 experiment and 50 percent from the site 7 experiment. The amount of gas-combustion crude that is accounted for by these two light fractions is 25 percent. The decrease in heavy gas oil and residuum contents of their crude compared to gas-combustion crude is greater than the decrease noted when our in situ heavy gas oil and residuum contents are compared to gas combustion.

TABLE 8. - Comparison of distillate yields from noncombustion in situ retorting, combustion in situ retorting, and gas combustion retorting

Retorting method	Fraction, vol pct of crude				Residuum
	Naphtha	Light distillate	Light gas oil	Heavy gas oil	
Hill, noncombustion in situ	40	40	15	4	1
BuMines, combustion in situ-4	14	46	25	7	8
BuMines, combustion in situ-7	10	40	30	13	7
BuMines, gas combustion	6	19	25	27	23

Table 9 lists the hydrocarbon-type analyses for the light distillates from each of these same crude oils. There is a greater difference noted when the saturate/olefin ratio for gas-combustion oil is compared to that ratio for the Hill's oil than when that ratio is compared for gas-combustion oil and our in situ oils. The distillate yield and hydrocarbon-type data from Hill's in situ crude indicate that the absence of oxygen in the retort gas may be partly responsible for the high naphtha and high saturate contents. Also responsible, as shown by our work, are the parameters involving shale size, retorting (combustion-zone) rate, and effective retorting temperature.

TABLE 9. - Comparison of light-distillate hydrocarbon-type compositions from noncombustion in situ retorting, combustion in situ retorting, and gas combustion retorting

Retorting method	Hydrocarbon type, vol pct		
	Saturates	Olefins	Aromatics
Hill, noncombustion in situ	64	5	31
BuMines, combustion in situ-4	55	15	30
BuMines, combustion in situ-7	52	22	28
BuMines, gas combustion	31	41	28

The concentration of aromatics in Hill's light distillate and of all of the light distillates studied in this work have a limited spread (tables 7 and 9), suggesting that the production of aromatics in this boiling range is independent of shale size, retorting rate, effective retorting temperature, and retort-gas composition.

One respect in which Hill's low-temperature oil does vary from our in situ oils is in the nitrogen content. Hill's crude had a nitrogen content of 0.8 percent, whereas the average for the six oils

in this study was 1.5 percent, or nearly twice as much. Hill gives the following explanation for his low nitrogen value:

"In the high temperature processes all of the kerogen undergoes decomposition. The nitrogen atoms become an integral part of the polymer and the thermal decomposition of this polymer gives products containing this nitrogen well distributed among the final product molecules.

Based on the experimental results we have concluded that the nitrogen in the kerogen is present in molecules of very high molecular weight which tend to remain in the shale at the decomposition temperatures below 800°F."

If the big difference in the two in situ retorting systems is the presence of air or the absence of methane, then it seems that nitrogen concentration in the crude oil is dependent more upon the retort-gas-composition parameter than on the other retorting parameters. It is either the presence of the methane or absence of oxygen that reduces the incorporation of nitrogen in the resulting shale oil.

Qualitatively speaking with respect to distillate yield and distillate composition, we can say that Hill's noncombustion in situ oil is similar to our combustion in situ oils. Quantitatively speaking we can say that the retorting parameters that control the character of the combustion in situ oils are the same parameters that largely control the character of the noncombustion in situ oil. These parameters are shale size, combustion-zone velocity, and effective retorting temperature.

SUMMARY

Differences in properties are noted when crude oils produced by rapidly retorting small-size (less than 3 inch) oil shale in aboveground retorts are compared with crude oils produced by slowly retorting large-size, mine-run shale in aboveground retorts. Significant extensions of these differences are noted when the latter oils are compared to in situ oils. For example, the specific gravity decreases from 0.94 to 0.92 to 0.89; the pour point decreases from 80° F to 60° F to 40° F; and the 100° F viscosity decreases from 400 SUS to 100 SUS to 65 SUS. Compositional changes are also noted; for example, the content of light distillate in the crude oil increases from 20 percent to 30 percent to 45 percent, while the residuum decreases from 20 percent to 7 percent to 6 percent. Another change is noted in the hydrocarbon-type distributions in the naphthas and light distillates. For example, the concentration of saturates in the light distillate increases from 30 to 40 to 50 volume percent while the olefins decrease from 40 to 30 to 20. The concentration of the aromatics in all of the light distillates is constant at about 30 volume percent.

When these compositional data are considered together with the oil yields from the aboveground retorts, the quantities of naphtha, of light distillate, of light gas oil, and of heavy gas oil each remain about the same. Expressed as gallons of each fraction per ton of 30-gpt shale, the average for each fraction is 1, 6, 6, and 7 respectively. However, the quantity of residuum from the small shale is considerably greater than from the mine-run shale. These figures are 6 and 1 respectively. Thus most of the decrease seen in recovery of oil from the aboveground retorting of mine-run shale compared with small shale is in the recovery of the residuum fraction. Because no increase in light materials accompanies the decrease in residuum, it is obvious that the loss of residuum is because the residuum is burned in those experiments that utilize large, mine-run oil shale.

The constancy of quantity of light products from the aboveground retorts can be used to estimate the oil recovery of the in situ process. For the two combustion in situ experiments, these recoveries were estimated to be 10.9 gpt and 13.1 gpt of 30-gpt shale.

Differences in characteristics between combustion in situ oil and the noncombustion in situ oil described by Hill and coworkers, such as olefin and nitrogen contents, could be due to the presence or absence of an oxidative atmosphere during retorting. Those oil characteristics that are the same for both combustion in situ oil and noncombustion in situ oil and that are different for these two in situ oils compared to oil produced by rapidly retorting small raw shale are due to the retorting parameters of shale size, combustion-zone velocity, and effective retorting temperature. These latter characteristics include distillate contents of the crude and hydrocarbon-type distribution within distillates.

ACKNOWLEDGMENTS

The work upon which this report is based was done under a cooperative agreement between the Bureau of Mines, U.S. Department of the Interior, and the University of Wyoming.

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